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# Summary of Major Changes Made to the Multi-Media, Multi-Concentration, Organic Analytical Service for Superfund (OLM04.3 to SOM01.1)

Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) (5102G)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to EPA at such sites are a series of volatile, semivolatile, pesticide, and Aroclor compounds that are analyzed using Gas Chromatography coupled with Mass Spectrometry (GC/MS) and Gas Chromatography with an Electron Capture Detector (GC/ECD). The Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technology Innovation (OSRTI) offers an analytical service that provides data from the analysis of water and soil/sediment samples for organic compounds for use in the Superfund decision-making process. Through a series of standardized procedures and a strict chain-of-custody, the low concentration organic analytical service produces data of known and documented

quality. This service is available through the Superfund Contract Laboratory Program (CLP).

#### **OVERVIEW OF MAJOR CHANGES**

This new organic analytical service provides a technical and contractual framework for laboratories to apply EPA/CLP analytical methods for the preparation, detection, and quantitative measurement of 52 volatile, 67 semivolatile, 21 pesticide, and 9 Aroclor target compounds in water samples.

- The pesticide and Aroclor analytical methods have been divided into separate exhibits.
- ii. A new method for the analysis of trace volatile target compounds has been added to SOM01.1.
- iii. The number of volatile and semivolatile compounds has been modified to include three new volatile compounds and two new semivolatile compounds. Also, the Xylene target compound has been broken into o-Xylene and m, p-Xylene. The compounds that have been added are detailed in the **Modifications to Target Compounds** section and are marked with a double asterisk (\*\*) in **Table 1**.
- iv. SOM01.1 includes Selected Ion Monitoring (SIM) analysis, which can be used to analyze certain trace volatile and semivolatile compounds. For a

- complete list of these compounds, please refer to **Table 1**, Target Compound List and Contract Required Quantitation Limits (CRQLs) for SOM01.1.
- v. SOM01.1 includes the requirement for the Staged Electronic Data Deliverable (SEDD) format for electronic deliverables. SEDD contains data elements arranged hierarchically by data nodes (parent elements), and consists of an Extensible Markup Language (XML) file(s) compliant with the XML specification 1.0 of the World Wide Web Consortium (W3C). The deliverable must be well-formed based on the W3C XML specification and must be valid based on the Document Type Definition (DTD).

## MODIFICATION TO TARGET COMPOUNDS

Since the CLP began in 1980, compounds have been added and removed from the Target Compound List (TCL) based on advances in analytical methods, evaluation of method performance data, and to meet the needs of Superfund program participants.

Compounds that have been added to the SOW are marked with a double asterisk (\*\*) in **Table 1**.

Table 1. Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) for SOM01.1\*

	Quantitation Limits					Quantitation Limits					
VOLATILES	Trace Water by SIM (µg/L)	Trace Water (µg/L)	Low Water (µg/L)	Low Soil (µg/kg)	Med. Soil (μg/kg)	VOLATILES (CON'T)	Trace Water by SIM (µg/L)	Trace Water (µg/L)	Low Water (µg/L)	Low Soil (µg/kg)	Med. Soil (μg/kg)
1. Dichlorodifluoromethane		0.50	5.0	5.0	250	44. Bromoform		0.50	5.0	5.0	250
2. Chloromethane		0.50	5.0	5.0	250	45. Isopropylbenzene		0.50	5.0	5.0	250
3. Vinyl chloride		0.50	5.0	5.0	250	46. 1,1,2,2-Tetrachloroethane		0.50	5.0	5.0	250
4. Bromomethane		0.50	5.0	5.0	250	47. 1,3-Dichlorobenzene		0.50	5.0	5.0	250
5. Chloroethane		0.50	5.0	5.0	250	48. 1,4-Dichlorobenzene		0.50	5.0	5.0	250
6. Trichlorofluoromethane		0.50	5.0	5.0	250	49. 1,2-Dichlorobenzene		0.50	5.0	5.0	250
7. 1,1-Dichloroethene		0.50	5.0	5.0	250	50. 1,2-Dibromo-3-chloropropane	0.050	0.50	5.0	5.0	250
8. 1,1,2-Trichloro-1,2,2-trifluoroethane		0.50	5.0	5.0	250	51. 1,2,4-Trichlorobenzene		0.50	5.0	5.0	250
9. Acetone		5.0	10	10	500	52. 1,2,3-Trichlorobenzene**		0.50	5.0	5.0	250
10. Carbon disulfide		0.50	5.0	5.0	250		Low				
11. Methyl acetate		0.50	5.0	5.0	250		Water		Low		
12. Methylene chloride		0.50	5.0	5.0	250		by SIM	Water	Soil by SIM	Low Soil	Med. Soil
13. trans-1,2-Dichloroethene		0.50	5.0	5.0	250	<b>SEMIVOLATILES</b>	(μg/L)	(μg/L)	(µg/kg)	(µg/kg)	(µg/kg)
14. Methyl tert-butyl ether		0.50	5.0	5.0	250	53. Benzaldehyde		5.0		170	5000
15. 1,1-Dichloroethane		0.50	5.0	5.0	250	54. Phenol		5.0		170	5000
16. cis-1,2-Dichloroethene		0.50	5.0	5.0	250	55. Bis(2-chloroethyl) ether		5.0		170	5000
17. 2-Butanone		5.0	10	10	500	56. 2-Chlorophenol		5.0		170	5000
18. Bromochloromethane**		0.50	5.0	5.0	250	57. 2-Methylphenol		5.0		170	5000
19. Chloroform		0.50	5.0	5.0	250	58. 2,2'-Oxybis (1-chloropropane)		5.0		170	5000
20. 1,1,1-Trichloroethane		0.50	5.0	5.0	250	59. Acetophenone		5.0		170	5000
21. Cyclohexane		0.50	5.0	5.0	250	60. 4-Methylphenol		5.0		170	5000
22. Carbon tetrachloride		0.50	5.0	5.0	250	61. N-Nitroso-di-n propylamine		5.0		170	5000
23. Benzene		0.50	5.0	5.0	250	62. Hexachloroethane		5.0		170	5000
24. 1,2-Dichloroethane		0.50	5.0	5.0	250	63. Nitrobenzene		5.0		170	5000
25. 1,4-Dioxane**	2.0	20	100	100	5000	64. Isophorone		5.0		170	5000
26. Trichloroethene		0.50	5.0	5.0	250	65. 2-Nitrophenol		5.0		170	5000
27. Methylcyclohexane		0.50	5.0	5.0	250	66. 2,4-Dimethylphenol		5.0		170	5000
28. 1,2-Dichloropropane		0.50	5.0	5.0	250	67. Bis(2-chloroethoxy) methane		5.0		170	5000
29. Bromodichloromethane		0.50	5.0	5.0	250	68. 2,4-Dichlorophenol		5.0		170	5000
30. cis-1,3-Dichloropropene		0.50	5.0	5.0	250	69. Napthalene	0.10	5.0	3.3	170	5000
31. 4-Methyl-2-pentanone		5.0	10	10	500	70. 4-Chloroaniline		5.0		170	5000
32. Toluene		0.50	5.0	5.0	250	71. Hexachlorobutadiene		5.0		170	5000
33. trans-1,3-Dichloropropene		0.50	5.0	5.0	250	72. Caprolactam		5.0		170	5000
34. 1,1,2-Trichloroethane		0.50	5.0	5.0	250	73. 4-Chloro-3-methylphenol		5.0		170	5000
35. Tetrachloroethene		0.50	5.0	5.0	250	74. 2-Methylnapthalene	0.10	5.0	3.3	170	5000
36. 2-Hexanone		5.0	10	10	500	75. Hexachlorocyclopentadiene		5.0		170	5000
37. Dibromochloromethane		0.50	5.0	5.0	250	76. 2,4,6-Trichlorophenol		5.0		170	5000
38. 1,2-Dibromoethane	0.050	0.50	5.0	5.0	250	77. 2,4,5-Trichlorophenol		5.0		170	5000
39. Chlorobenzene		0.50	5.0	5.0	250	78. 1,1'-Biphenyl		5.0		170	5000
40. Ethylbenzene		0.50	5.0	5.0	250	79. 2-Chloronapthalene		5.0		170	5000
41. o-Xylene**		0.50	5.0	5.0	250	80. 2-Nitroaniline		10		330	10000
42. m, p-Xylene**		0.50	5.0	5.0	250	81. Dimethylphthalate		5.0		170	5000
43. Styrene		0.50	5.0	5.0	250	82. 2,6-Dinitrotoluene		5.0		170	5000

<sup>\*</sup> For volatile medium soils, quantitation limits are approximately 50 times the quantitation limits for low soils. For semivolatile medium soils, quantitation limits are approximately 50 times the quantitation limits for low soils.

\*\* This is a newly-added target compound.

Table 1. Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) for SOM01.1\* (Con't)

		Qı	ıantitation I	Limits		Quantitation Limits			
SEMIVOLATILES (CON'T)	Low Water by SIM (µg/L)	Water (µg/L)	Low Soil by SIM (µg/kg)	Low Soil (µg/kg)	Med. Soil (µg/kg)	PESTICIDES	Water (μg/L)	Soil (µg/kg)	
83. Acenaphthylene	0.10	5.0	3.3	170	5000	120. alpha-BHC	0.050	1.7	
84. 3-Nitroaniline		10		330	10000	121. beta-BHC	0.050	1.7	
85. Acenaphthene	0.10	5.0	3.3	170	5000	122. delta-BHC	0.050	1.7	
86. 2,4-Dinitrophenol		10		330	10000	123. gamma-BHC (Lindane)	0.050	1.7	
87. 4-Nitrophenol		10		330	10000	124. Heptachlor	0.050	1.7	
88. Dibenzofuran		5.0		170	5000	125. Aldrin	0.050	1.7	
89. 2,4-Dinitrotoluene		5.0		170	5000	126. Heptachlor epoxide	0.050	1.7	
90. Diethylphthalate		5.0		170	5000	127. Endosulfan I	0.050	1.7	
91. Fluorene	0.10	5.0	3.3	170	5000	128. Dieldrin	0.10	3.3	
92. 4-Chlorophenyl-phenylether		5.0		170	5000	129. 4,4'-DDE	0.10	3.3	
93. 4-Nitroaniline		10		330	10000	130. Endrin	0.10	3.3	
94. 4,6-Dinitro-2-methylphenol		10		330	10000	131. Endosulfan II	0.10	3.3	
95. N-Nitrosodiphenylamine		5.0		170	5000	132. 4-4'-DDD	0.10	3.3	
96. 1,2,4,5-Tetra chlorobenzene**		5.0		170	5000	133. Endosulfan sulfate	0.10	3.3	
97. 4-Bromophenyl-phenylether		5.0		170	5000	134. 4-4'-DDT	0.10	3.3	
98. Hexachlorobenzene		5.0		170	5000	135. Methoxychlor	0.50	17	
99. Atrazine		5.0		170	5000	136. Endrin ketone	0.10	3.3	
100. Pentachlorophenol	0.20	10	6.7	330	10000	137. Endrin aldehyde	0.10	3.3	
101. Phenanthrene	0.10	5.0	3.3	170	5000	138. alpha-Chlordane	0.050	1.7	
102. Anthracene	0.10	5.0	3.3	170	5000	139. gamma-Chlordane	0.050	1.7	
103. Carbazole		5.0		170	5000	140. Toxaphene	5.0	170	
104. Di-n-butylphthalate		5.0		170	5000				
105. Fluoranthene	0.10	5.0	3.3	170	5000	AROCLORS	Water (µg/L)	Soil (µg/kg)	
106. Pyrene	0.10	5.0	3.3	170	5000	141. Aroclor-1016	1.0	33	
107. Butylbenzylphthalate		5.0		170	5000	142. Aroclor-1221	1.0	33	
108. 3,3'-Dichlorobenzidine		5.0		170	5000	143. Aroclor-1232	1.0	33	
109. Benzo (a) anthracene	0.10	5.0	3.3	170	5000	144. Aroclor-1242	1.0	33	
110. Chrysene	0.10	5.0	3.3	170	5000	145. Aroclor-1248	1.0	33	
111. Bis(2-ethylhexyl) phthalate		5.0		170	5000	146. Aroclor-1254	1.0	33	
112. Di-n-octylphthalate		5.0		170	5000	147. Aroclor-1260	1.0	33	
113. Benzo (b) fluoroanthene	0.10	5.0	3.3	170	5000	148. Aroclor-1262**	1.0	33	
114. Benzo (k) fluoroanthene	0.10	5.0	3.3	170	5000	149. Aroclor-1268**	1.0	33	
115. Benzo (a) pyrene	0.10	5.0	3.3	170	5000				
116. Indeno (1,2,3-cd)-pyrene	0.10	5.0	3.3	170	5000				
117. Dibenzo (a,h)-anthracene	0.10	5.0	3.3	170	5000				
118. Benzo (g,h,i) perylene	0.10	5.0	3.3	170	5000				
119. 2,3,4,6-Tetrachlorophenol**		5.0		170	5000				

<sup>\*</sup> For volatile medium soils, quantitation limits are approximately 50 times the quantitation limits for low soils. For semivolatile medium soils, quantitation limits are approximately 30 times the quantitation limits for low soils.

<sup>\*\*</sup> This is a newly-added target compound.

The following modifications were made to the organic analytical service TCL:

- i. New volatile compounds include: Bromochloromethane, 1,4-Dioxane, and 1,2,3-Trichlorobenzene. The Xylene target compound was separated into o-Xylene and m, p-Xylene.
- ii. New semivolatile compounds include: 1,2,4,5-Tetra chlorobenzene and 2,3,4,6-Tetrachlorophenol.
- iii. New Aroclors include: Aroclor-1262 and Aroclor-1268.

#### MODIFICATIONS TO METHODS

Current methods have been modified in an effort to allow CLP participants to use newer techniques or address previous issues. The following items summarize the modifications to the organic analytical service:

- The trace volatiles method has been added to this service.
- SIM analysis can be used to analyze certain trace volatiles and semivolatile compounds.
- Matrix spiking solutions may be prepared monthly instead of weekly, or sooner if the solution has degraded or evaporated.
- Method Detection Limit (MDL) determination requirements have been incorporated into the method.

# Trace Volatiles

The new trace volatiles method includes:

- i. For trace volatile water samples, 25 mL of water sample is added to a purge and trap device and purged with an inert gas at room temperature (or higher if all technical acceptance criteria is met for all standards, samples, and blanks). The volatiles purged from the sample are trapped on a solid sorbent. They are subsequently desorbed by rapidly heating the sorbent and then introduced into a GC/MS system.
- ii. Requirements for SIM analysis are incorporated into this method.
- iii. The Deuterated Monitoring Compounds (DMCs) have been updated, replacing Bromoform-d with 1,4-dioxane-d8. Additionally, the DMC recovery limits have been modified.
- iv. The Percent Relative Standard Deviation (%RSD) and the Percent Difference (%D) QC limits for DMCs have been added for Initial Calibration (ICAL) and Continuing Calibration Verification (CCV) standards, respectively.

v. A closing CCV that meets the technical acceptance criteria is now required.

#### Volatiles

The following items were changed for volatiles:

- Low-level soil dilutions will no longer be permitted since this procedure could lead to loss of analytes. In cases where an analyte exceeds the calibration range for a soil sample prepared by the low-level procedure, the soil sample must be prepared and analyzed by the medium-level procedure.
- Analysis of unpreserved, unfrozen soil samples for volatiles analysis must be completed within 24 hours of Validated Time of Sample Receipt (VTSR).
- iii. Requirements for SIM analysis have been incorporated into the method.
- iv. DMCs have been added to replace surrogates.
- v. A closing CCV that meets the technical acceptance criteria is now required.

### **Semivolatiles**

The following items were changed for semivolatiles:

- i. Requirements for SIM analysis have been incorporated into the method.
- ii. DMCs have been added to replace surrogates.
- iii. A closing CCV that meets the technical acceptance criteria is now required.

#### Pesticides

The following items were changed for pesticides:

- i. The pesticides analytical method was split from Aroclors into a separate exhibit.
- ii. A five-point ICAL standard is now required for this method.
- iii. The analysis of a Laboratory Control Sample (LCS) is now required for this method.
- iv. Additional GC column options have been incorporated into the method.

# Aroclors

The following items were changed for Aroclors:

- i. The Aroclors analytical method was split from pesticides into a separate exhibit.
- ii. Acid cleanup is now required for sample extract cleanup.
- iii. The analysis of an LCS is now required for this method.
- iv. A five-point initial calibration is now required for Aroclor-1216 and Aroclor-1260. A single-point initial calibration is required for other Aroclors; however, if detected, then a five-point calibration is required.
- v. Additional GC column options have been incorporated into the method.

# **CONTACTING EPA**

For more information, or for suggestions to improve this analytical service, please contact:

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